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Preliminary Screening of Palm-Frond-Derived Sodium Carboxymethyl Cellulose as a Rheology and Filtration Modifier for Water-Based Drilling Fluids

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Abstract

Objective: This preliminary screening study evaluated sodium carboxymethyl cellulose synthesized from palm-frond cellulose as a locally sourced modifier for bentonite-based water-based drilling fluids. **Methodology:** Palm-frond cellulose was isolated by repeated alkaline cooking and four hypochlorite-bleaching cycles, carboxymethylated with monochloroacetic acid, purified, and screened against commercial CMC. Single, unreplicated measurements of rotational rheology and 30-min filtration were obtained; therefore, no uncertainty estimates, error bars, confidence intervals, p-values, or claims of statistical equivalence are reported. Bentonite fluids contained 22.5 g of clay in 350 mL of water and were tested immediately after mixing rather than after a 16-h hydration period. Short temperature conditioning from 25˚C to 90˚C was also used and was not equivalent to 16-h hot rolling. **Results:** The synthesized material showed low-viscosity-grade behavior at the test conditions and required a higher mass dosage than the commercial material. At 25˚C, increasing the palm-frond material from 0 to 25% of bentonite mass raised the 600-rpm reading from 25 to 38 and reduced filtrate from 19 to 10 mL/30min. At 50˚C, isolated filtrate readings were 5.8 and 6.0 mL for the palm-frond and commercial systems, respectively; this 0.2-mL difference is not interpreted because repeatability was not quantified. In a KCl-polymer screen, 4 g palm-frond material increased the yield point from 2 to 6 lb/100ft^2 and reduced filtrate from 17.2 to 15.6 mL, but did not match the PAC-LV reference. **Conclusion:** The results establish feasibility only. Replicated testing, controlled aging, complete compositional characterization, and hot-rolling validation are required before comparative or field-performance claims can be made.

Keywords

Carboxymethyl Cellulose, Palm Fronds, Water-Based Drilling Fluid, Bentonite, Rheology, Fluid Loss, Biomass Valorization

1. Introduction

Water-based drilling fluids remain central to well construction because they can provide pressure control, cuttings transport, cooling, lubrication, and wellbore support with a generally lower environmental burden than nonaqueous systems. Their performance, however, depends on maintaining a narrow rheological and filtration envelope as clay hydration, drilled solids, salinity, and temperature change. Excessive high-shear viscosity increases circulating pressure losses, whereas insufficient low-shear structure compromises hole cleaning and suspension. Excess filtrate and thick filter cake can amplify differential sticking, torque, and drag, and near-wellbore impairment.

Sodium carboxymethyl cellulose (CMC) is an anionic, water-soluble cellulose ether used in drilling fluids as a viscosifier and fluid-loss reducer. Adsorption of polymer segments on clay surfaces, hydration of carboxymethyl groups, chain entanglement, and pore bridging within the filter cake jointly modify flow and filtration. The response depends strongly on molecular mass, degree and uniformity of substitution, purity, salinity, temperature, and the clay-polymer ratio. Accordingly, commercial CMC is supplied in low- and high-viscosity grades, and material qualification is performance-based under API Specification 13A rather than inferred from chemical identity alone [1]. Polymer-clay suspensions commonly exhibit shear-thinning and yield-stress behavior that is represented more realistically by the Herschel-Bulkley model than by a two-point Bingham approximation [2]-[5].

Agricultural residues offer renewable feedstocks for drilling-fluid additives and may reduce dependence on imported chemicals. Recent studies have reported biomass-derived polymers, direct bio-additives, palm-frond-derived CMC, proteinaceous biomass additives, and biodegradable materials for controlling water-based drilling-fluid filtration under ambient and elevated-temperature conditions [6]-[12]. In particular, Reference [10] already demonstrated synthesis and drilling-fluid application of palm-frond CMC. The present work is not claimed as the first palm-frond CMC study; its narrower contribution is a preliminary, side-by-side dosage and temperature screening against a commercial CMC in a selected bentonite, together with an exploratory substitution test in a KCl-polymer formulation. This distinction also limits the conclusions to the specific, unreplicated conditions examined.

This preliminary study, therefore, aimed to 1) document the available synthesis record and its reproducibility gaps; 2) screen the rheological and filtration response of the palm-frond product and commercial CMC in bentonite suspensions

over 25˚C - 90˚C; 3) report the three-clay selection data; and 4) explore the product in a KCl-polymer formulation. Because the archived dataset contains single measurements, the analysis emphasizes trends and limitations and does not test statistical significance or equivalence.

2. Methodology and Experimental Setup

2.1. CMC Synthesis and Materials

Palm fronds collected in Khartoum State were washed, air-dried, ground, and soaked in water for approximately 20 min. The available experimental record states that the fibers were cooked repeatedly in 500 mL of 1 M NaOH (approximately 4% w/v) at 80˚C for 4 h and washed with distilled water. Bleaching was then repeated four times for 4 h at 80˚C using a commercial sodium hypochlorite solution reported as 1.7% NaOCl, with 2.5% NaOH and 75 mL acetic acid, followed by washing until hypochlorite odor was no longer detected. However, the record does not report the dry palm-frond mass treated in each batch; consequently, the solid-to-liquid ratio cannot be reconstructed. It also does not report measured pH values or a controlled pH trajectory during cooking or bleaching.

For carboxymethylation, 2 g of isolated cellulose was suspended in 40 mL of an alcohol medium, and 10 mL of 30% aqueous NaOH was added dropwise over 30 min before 1 h of mixing at room temperature. The archived method lists isopropanol, n-butanol, ethanol, and methanol as candidate alcohols but does not identify which single solvent or mixture, its purity, or its composition was used for the reported batch. Likewise, 6.00 g monochloroacetic acid was dissolved in 10 mL of a solvent listed as isopropanol or ethanol, without identifying the selected solvent or purity. The reaction proceeded at 55˚C for 4 h, was neutralized with 90% acetic acid, filtered, washed five times with 70% ethanol, and dried at 60˚C. Purification comprised dispersion in 60 mL of 95% ethanol, addition of 10 mL of 2 M nitric acid, heating to boiling for 5 min, further agitation for 15 min, washing with 95% ethanol and hot 80% ethanol, methanol washing, and drying at 105˚C for 3 h. These solvent ambiguities are now reported explicitly because they prevent exact reproduction and should be resolved in a controlled repeat study.

Cellulose yield, final CMC yield, moisture, ash, chloride, and active-polymer content were not measured in the archived work. Degree of substitution and functional-group confirmation by FTIR, NMR, or titration were also not available. Accordingly, the material is described operationally as a palm-frond-derived CMC candidate, and XRD similarity is not treated as proof of etherification, substitution level, or substitution uniformity.

A commercial high-viscosity CMC served as the benchmark. Three bentonites (A, B, and C) were screened using 22.5 g of clay in 350 mL of deionized water. Each clay suspension was mixed for 5 min and tested without a reported 16-h prehydration/aging period. Bentonite B was selected because it combined the lowest PV (7 cP) with a moderate YP (11 lb/100ft^2), although its filtrate (18.8 mL) was the highest of the three. The full screening dataset is provided in **Table 1**, because

fresh and aged clay dispersions can give materially different rheology and filtration. All subsequent concentration results therefore apply to freshly prepared, non-aged bentonite B under the recorded protocol.

Table 1. Screening data for the three candidate bentonites (single measurements; 22.5 g/350mL).

Property	Bentonite A	Bentonite B	Bentonite C
$\theta 3/\theta 6$	5/10	7/10	5/7
$\theta 100/\theta 200$	16/21	13/16	10/12
$\theta 300/\theta 600$	27/37	18/25	15/24
PV (cP)	10	7	9
YP (lb/100 ft^2)	17	11	6
Gel, 10 s/10min	5/37	9/35	5/34
Density (lb/gal)	8.7	8.65	8.7
Marsh funnel (s)	47	42	36
pH	8	8	8
Filtrate (mL)	15	18.8	14

2.2. Characterization and Fluid Testing

Powder X-ray diffraction was conducted with a Philips X'Pert diffractometer using Cu Kα radiation (λ = 1.5406 Å). The patterns were used only for qualitative comparison. The phase search for the synthesized product did not converge under Rietveld refinement and showed substantial residual inorganic peaks. XRD, therefore, neither confirms carboxymethyl ether formation nor quantifies the degree of substitution, substitution uniformity, active content, or polymer purity. These properties remain unmeasured and are required for definitive identification and structure-performance interpretation.

Polymer-containing fluids were mixed for 20 min and then evaluated using a ZNN-D6 six-speed rotational viscometer. The mixer model was recorded, but its rotational speed was not; this is a reproducibility limitation. No additional hydration period or 16-h aging step was documented after polymer addition. Apparent viscosity (AV), plastic viscosity (PV), and yield point (YP) were calculated as AV = $\theta 600/2$, PV = $\theta 600 - \theta 300$, and YP = $\theta 300 - $ PV. Filtration was measured for 30 min at 0.69 MPa. Commercial CMC additions were 0.1 - 2.0 g (0.44% - 8.89% of bentonite mass), whereas palm-frond material additions were 1.1 - 5.6 g (5% - 25%). For the temperature series, samples were conditioned for 30 min at 25˚C, 50˚C, 75˚C, or 90˚C. The archived record does not establish whether evaporation was controlled, whether the viscometer cup was maintained at the stated temperature, or whether dial readings were taken hot or after cooling. Consequently, the temperature data are reported as screening observations, not intrinsic thermal-stability measurements. Short static conditioning is not equivalent to sealed 16-h hot rolling.

Grade screening was operational rather than a complete API 13A qualification. The commercial material was screened at 2.2 g in 350 mL deionized water after 20 min mixing and gave $\theta 600 = 37$. The palm-frond material was screened separately at 10.5 g in 350 mL deionized water after 20 min mixing and gave $\theta 600 = 30$. Because the concentrations were non-equivalent and the complete prescribed API solution matrix was not run, these readings support only the descriptive labels "higher-viscosity commercial material" and "lower-viscosity palm-frond material" under the study conditions.

Every reported condition represents one measurement (n = 1). No laboratory duplicates, standard deviations, confidence intervals, or p-values are available. Error bars cannot be calculated retrospectively. Differences close to normal test repeatability, including 5.8 versus 6.0 mL filtrate, are therefore not interpreted as meaningful differences (Table 2).

Table 2. Experimental design and measured responses.

Variable	Commercial CMC	Palm-Frond CMC
Grade by 600-rpm screening	High viscosity	Low viscosity
Dosage in 22.5 g of bentonite	0.1 - 2.0 g (0.44% - 8.89%)	1.1 - 5.6 g (5% - 25%)
Aqueous phase	350 mL deionized water	350 mL deionized water
Temperature	25˚C, 50˚C, 75˚C, 90˚C	25˚C, 50˚C, 75˚C, 90˚C
Responses	$\theta 600$, PV, YP, filtrate	$\theta 600$, PV, YP, filtrate

2.3. KCl-Polymer Formulation Screen

Three 350-mL KCl-polymer fluids were compared: a reference containing 4 g PAC-LV; the same formulation without PAC-LV; and the PAC-free formulation treated with 4 g palm-frond CMC. Each also contained 0.5 g caustic soda, 0.5 g soda ash, 0.5 g xanthan-type viscosifier, 2 g fluid-loss additive, and 15 g KCl. This sequence isolated the incremental contribution of the synthesized CMC, although it was a screening comparison rather than a statistically replicated replacement study.

3. Results and Discussion

3.1. Product Grading and Bentonite Response at 25˚C

Under the operational screening described in Section 2.2, the commercial material gave $\theta 600 = 37$ at 2.2 g/350mL, whereas the palm-frond material gave $\theta 600 = 30$ at 10.5 g/350mL. These non-equivalent concentrations do not support a formal, reproducible API grade assignment. They indicate only that the palm-frond product had substantially lower viscosity-building efficiency per unit mass in this dataset. Depending on the selected response target, approximately three to ten times more palm-frond material was required than commercial CMC.

Both products reduced fluid loss monotonically at 25˚C, but their rheological signatures were distinct (Figure 1). Commercial CMC strongly increased $\theta 600$ and

YP: at 8.89%, θ600 reached 197, and YP reached 141 lb/100ft^2. Such values indicate severe over-treatment for many circulating systems, despite the favorable filtrate of 8.8 mL. In contrast, palm-frond CMC increased θ600 gradually from 25 to 38 and PV from 7 to 15 cP over 0% - 25%, while YP decreased from 11 to 8 lb/100ft^2 and then remained nearly constant. This combination suggests that the synthesized grade primarily increased continuous-phase viscosity and filter-cake quality without generating the strong interparticle structure observed for the commercial high-viscosity grade.

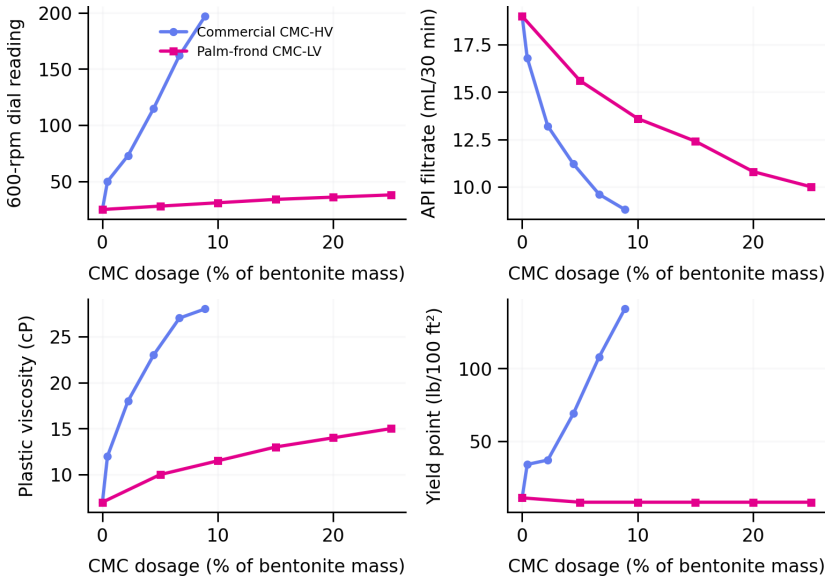

Figure 1. Concentration response at 25˚C. Percentages are relative to bentonite mass. Each point is a single measurement (n = 1); lines guide the eye, and no error bars are available.

At the highest tested dosage, palm-frond CMC reduced filtrate from 19 to 10 mL/30min, a 47.4% decrease. A 20% dosage delivered 10.8 mL with PV of 14 cP and YP of 8 lb/100ft^2. These values may be useful where filtration control is needed without a large increase in annular yield stress, but the relatively low YP also means that hole-cleaning performance cannot be assumed. The required concentration should therefore be selected jointly from hydraulic, cuttings-transport, and filtration constraints rather than from fluid loss alone (Table 3).

Table 3. Selected 25˚C performance points.

Fluid	CMC Dosage	θ600	PV (cP)	YP (lb/100 ft^2)	Filtrate (mL)
Base bentonite	0%	25	7	11	19
Commercial CMC	2.22%	73	18	37	13.2
Commercial CMC	6.67%	162	27	108	9.6
Palm-frond CMC	10%	31	11.5	8	13.6
Palm-frond CMC	20%	36	14	8	10.8
Palm-frond CMC	25%	38	15	8	10.0

3.2. Temperature Response

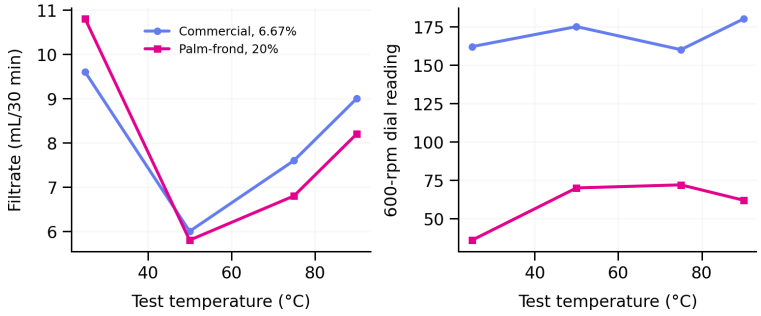

Figure 2. Short-duration temperature-screening results at fixed, non-equivalent dosages: commercial CMC at 6.67% and palm-frond material at 20% of bentonite mass. Each point is a single measurement (n = 1); no error bars are available, and the protocol was not equivalent to 16-h hot rolling.

At the selected dosages, the single filtrate readings decreased between 25˚C and 50˚C, reaching 6.0 mL for commercial CMC and 5.8 mL for the palm-frond material. The 0.2-mL separation is not evidence of superior or equivalent performance because repeatability was not measured, and no statistical comparison is possible. Above 50˚C, the isolated filtrate readings increased to 9.0 and 8.2 mL at 90˚C. Rheological responses were nonmonotonic: the palm-frond system increased from $\theta 600 = 36$ at 25˚C to 72 at 75˚C and then decreased to 62 at 90˚C. This unexpected increase may reflect continued hydration, evaporation, measurement-temperature differences, or experimental variation; the archived protocol cannot distinguish among these explanations (Figure 2).

The temperature series demonstrates only short-duration screening behavior. Evaporation control and the temperature at which dial readings were taken were not documented, and the samples were not subjected to sealed 16-h hot rolling followed by measurement at a common temperature. The results, therefore, do not establish thermal stability, equivalence, or superiority. Replicated, sealed aging with controlled cooling and mass-balance checks is required before temperature-dependent claims can be made.

3.3. KCl-Polymer Formulation Performance

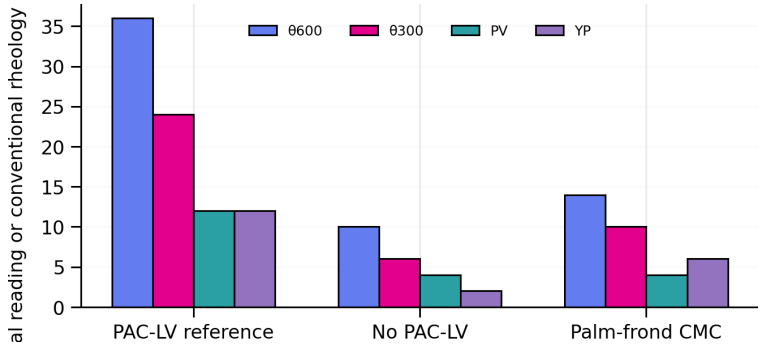

Figure 3. KCl-polymer rheology screen. $\theta 600$ and $\theta 300$ are dial readings; PV is in cP, and YP is in lb/100 ft^2. Values are single measurements (n = 1).

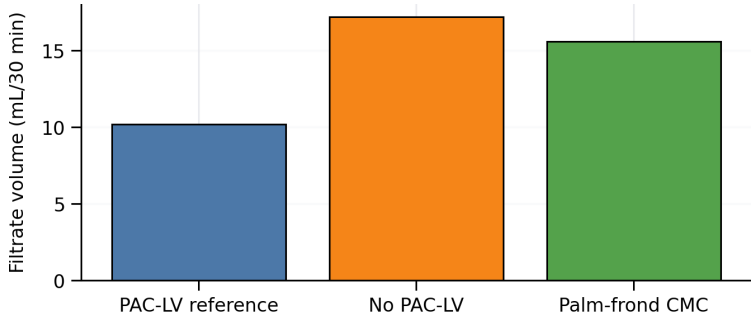

Figure 4. KCl-polymer filtration screen. Filtrate volume is shown separately to avoid combining unlike units on one axis. Values are single measurements (n = 1).

Removing PAC-LV from the KCl-polymer reference reduced θ600 from 36 to 10, PV from 12 to 4 cP, and YP from 12 to 2 lb/100ft^2, while the single filtrate reading increased from 10.2 to 17.2 mL (**Figure 3** and **Figure 4**). Addition of 4 g palm-frond material raised θ600 to 14 and YP to 6 lb/100ft^2 and produced a filtrate reading of 15.6 mL. These isolated values show directional recovery relative to the PAC-free formulation, but they are not evidence of a statistically significant 9.3% filtration improvement or of replacement equivalence. The formulation was an exploratory compatibility screen.

Foaming and incomplete dissolution were observed during mixing. Both can bias rheology and filtration by entraining air and reducing the effective dissolved-polymer concentration. Process optimization should therefore address particle size, salt removal, drying history, dissolution sequence, and defoamer compatibility. The strong NaCl signal reported by qualitative phase matching is consistent with incomplete inorganic removal, although XRD alone cannot determine polymer purity. Conductometric ash, chloride, moisture, degree of substitution, molecular-mass distribution, and FTIR/NMR characterization are required to connect synthesis chemistry with drilling performance (**Table 4**).

Table 4. KCl-polymer formulation screening results.

Formulation	θ600	θ300	PV (cP)	YP (lb/100 ft^2)	Filtrate (mL/30 min)	pH
Reference with 4 g PAC-LV	36	24	12	12	10.2	8.5
Without PAC-LV	10	6	4	2	17.2	8.5
With 4 g palm-frond CMC	14	10	4	6	15.6	9.0

3.4. Engineering Significance and Limitations

The most defensible application for the current product is as a low-viscosity filtration-control additive in fresh to moderately saline water-based fluids where excessive rheology is undesirable. The palm-frond route converts an agricultural residue into a technically active polymer and could reduce exposure to imported specialty chemicals. However, cost advantage cannot be inferred from customs values alone. A credible economic assessment must include cellulose yield, reagent recovery, solvent recycle, water and energy use, neutralization, drying, waste treat-

ment, quality-control losses, packaging, transport, and the higher field dosage required relative to commercial CMC or PAC.

Several limitations define the boundary of the conclusions. All conditions were measured once (n = 1), so variability and statistical significance are unknown. The two CMC materials were tested at different concentrations, preventing intrinsic efficiency or equivalence claims. Bentonite was tested without documented 16-h prehydration, mixer speed was unrecorded, and the temperature protocol did not document evaporation control or measurement temperature, and was not equivalent to hot rolling. Cellulose yield, CMC yield, active content, moisture, residual salt, degree of substitution, and functional-group confirmation were not measured. Qualitative XRD cannot establish etherification or substitution uniformity. Finally, the KCl formulation was a single screen without contamination, lubricity, shale-recovery, sag, or return-permeability testing. The study should therefore be read as feasibility screening only.

Future work should repeat the key conditions with at least three independent preparations, report mean ± standard deviation and confidence intervals, and apply statistical tests selected before data collection. Base bentonite should be prehydrated for a controlled period, and mixer speed, sequence, and temperature should be recorded. Thermal testing should use sealed 16-h hot rolling, controlled cooling to a common reporting temperature, evaporation/mass-balance checks, and replicate API/HPHT filtration. Synthesis records should identify the exact alcohol and monochloroacetic-acid solvent, their purities and compositions, solid-to-liquid ratio, pH, and cycle count. Cellulose and CMC yields, degree of substitution, FTIR/NMR evidence, molecular-mass distribution, moisture, ash, chloride, and active content should be reported before benchmarking against API 13A and commercial CMC-LV/PAC-LV.

4. Conclusions

The archived experiments provide preliminary evidence that a palm-frond-derived product can modify bentonite-fluid rheology and filtration; they do not provide complete chemical confirmation or a formal API grade assignment.

At 25˚C, the single measurements showed filtrate decreasing from 19 to 10 mL/30 min and PV increasing from 7 to 15 cP as palm-frond material increased to 25% of bentonite mass. These are unreplicated screening trends.

The isolated 5.8- and 6.0-mL readings at 50˚C are not statistically distinguishable because repeatability was not measured; no claim of equivalence or superiority is made.

In the KCl-polymer screen, the palm-frond treatment produced directional recovery relative to the PAC-free formulation but did not match the PAC-LV reference; the result requires replication.

Before field qualification, the synthesis must be fully specified and characterized, and drilling-fluid performance must be validated using replicated tests, controlled bentonite aging, quantified uncertainty, evaporation control, and sealed

16-h hot rolling.

Conflicts of Interest

The author declares no conflicts of interest regarding the publication of this paper.

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